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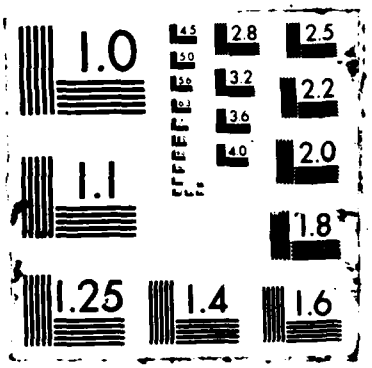
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This investigation examined the sintering enhancement of refractory metals and attempted to link activator effects to sintering kinetics, microstructure and properties. Work was carried out predominately on the W-Ni-Fe heavy alloy system. In addition, it was recognized that property improvement in heavy alloys could be made possible by understanding impurity-microstructure interactions, fracture behavior, and finally the combined effect of processing conditions and materials characteristics on densification during liquid phase sintering. This report addresses each of the above concerns and presents an overview of the work to date.

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PROPERTIES OF REFRACTORY METALS PROCESSED BY ENHANCED SINTERING TREATMENTS

FINAL REPORT

R. M. German and S. Farooq

May 1987

U. S. ARMY RESEARCH OFFICE

DAAG29-84-K-0026

RENSSELAER POLYTECHNIC INSTITUTE

The aim of this investigation was to specifically examine the sintering enhancement of refractory metals and to link activator effects to sintering kinetics, microstructure and properties. In addition, it was recognized that property improvement in heavy alloys could be made possible by understanding impurity-microstructure interactions, fracture behavior, and finally the combined effect of processing conditions and material characteristics on densification during liquid phase sintering. The studies undertaken during the past three years addressed the above problems. While periodic reports described the results from such individual studies, this final report will present an overview of the work carried out to date.

The effect of processing conditions on as-sintered properties was studied. Initial studies were devoted to optimizing the sintering and heat treatment cycle in order to avoid extraneous variations and inconsistencies in the results. The optimum cycles used are:

Sintering Cycle - heat at 10 K/min to sintering temperature (1733-1773 K), hold at temperature for 30-60 minutes in hydrogen. Argon was used in the last 10 minutes of the sintering cycle and during cooling. The samples were cooled at 3 K/min through solidification temperature, and furnace cooled in argon.

Heat Treatment Cycle - heat at 10 K/min to 1373 K in argon, hold for 1 hour and quench in water.

It was found that sintering temperature and time affect grain size, dihedral angle and contiguity. These in turn affect the fracture behavior and hence the mechanical properties of heavy alloys. The manner in which microstructural features relate to each other was modelled theoretically. It was shown by such calculations that the contiguity was directly related to volume fraction of matrix phase and the dihedral angle. Briefly, small volume fractions of matrix phase and large dihedral angles increase contiguity. It was also shown that increased contiguity could significantly reduce the ductility.

From the experimental data it was apparent that the volume fraction did not change with sintering temperature and time. However, the dihedral angle increased with a decrease in sintering temperature. The above microstructural features were correlated with mechanical properties.

Based on the results of such experiments, it was felt that a better understanding of fracture behavior of heavy alloys was required. It was found that cracks initiate at the grain boundaries. This process of initiation was limited to the surface layers due to the absence of a triaxial stress field at the sample surface. Tensile fracture of heavy alloys

is thus controlled by the accumulation of a critical amount of damage within the near surface region of the specimen. The first cracks appeared only at a few percent plastic strain and their frequency increased with an increase in tungsten content. The average crack size at a given plastic strain was found to be greater with lower tungsten content. A model was proposed in order to predict the fracture stress and strain in heavy alloys based upon microstructural characteristics. Fracture of a tensile bar is assumed to occur when a critical crack separation distance is reached within the near surface region. The crack separation distance depends upon both the number and size of the grain boundary cracks. These factors are determined by the strain, and microstructural variables such as volume fraction, grain size, dihedral angle, contiguity, etc. Based on the knowledge of true stress-strain behavior of these alloys, the proposed model can be used to predict mechanical properties.

It was also observed that the sintering atmosphere played an important role in determining densification behavior of these alloys. Water vapor evolution during sintering in a dry hydrogen atmosphere can lead to excessive porosity which is detrimental to heavy alloy properties. The effect of a pre-reduction treatment was shown to benefit in such cases. In the case of powders with high oxygen contents, the use of a wet hydrogen atmosphere in the presence of a liquid prevented the evolution of water vapor, resulting in high densities in the as-sintered product. Optimum processing of heavy alloys was thus shown to require careful dewpoint control.

Theoretical modelling work had demonstrated that the sintering atmosphere controlled densification in the final stage of liquid phase sintering, and that a high diffusivity gas or vacuum sintering would give rise to rapid densification and hence higher as-sintered densities. Experiments using a vacuum resulted in excellent properties in the as-sintered material, generally exceeding those of conventionally sintered material.

Marginal improvements in heavy alloy properties can be obtained by changing the segregation patterns within these alloys. Theoretical modelling and experimental results clearly demonstrated that limited amount of segregation of elements like carbon, boron and nitrogen at interfaces would improve interface strengths and ductility. This improvement can be attributed both to a direct increase in bond strengths and indirectly to competitive segregation. It was also shown that the segregation of sulfur, phosphorus and oxygen resulted in reduced properties. Selective segregation can be successfully used to obtain substantial property improvement, particularly when the base level impurity concentration is far from optimal.

The final part of this investigation was devoted to the study of liquid phase sintering in heavy alloys and the influence of material and processing parameters on densification. The variables studied were solubility of the major phase (W) in the additive, heating rate to sintering temperature and additive homogeneity. It has been shown that densification before and after the formation of the liquid phase is a strong function of all of the above parameters. Detailed theoretical and experimental studies are currently underway to better understand this effect.

In summary, this investigation has provided valuable insight into the processes and mechanisms that control densification behavior, microstructure, fracture and properties of heavy alloys. The effort has resulted in the development of optimal processing methods, theoretical models for prediction of sintering and mechanical behavior, and the ability to control properties with a great degree of consistency. In general it was shown that the final properties of heavy alloys are extremely structure sensitive, which is in turn related to the processing history.

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